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Oh well I will describe some of my experiences with PETN…

I didn't document some of my very first PETN synths, but I managed to documentate most of them.

I followed powerlabs's procedure I didn't recrystalize it tho... No pictures only some pretty crappy video.
I used the whole product (~20grams) and 3grams of HMTD for detonator:
http://www.youtube.com/watch?v=YdVKhokiinE
Notice the launchramp behind charge? It was meant to be rocket launched charge but my friend's ignition system for charge failed and rocket was very unstable(good thing it didn't detonate) I decided to detonate it on ground.

Ok about a year ago I made a paralel nitration:
2x10g PE, 34mL 65% HNO3 and 25mL of 96% H2SO4 in two 250mL beakers.
Picture of workplace: http://i242.photobucket.com/albums/f...o/IMG_2101.jpg

First I mixed the acids at below -10°C, then PE was added in small batches (most of the time ~1g of PE a time) while keeping temperature below 0°C. After adding all the PE I moved the beakers from salt/ice bath into warm water. Temperature of nitration mix was around 16°C I sustained this temperature for 30 minutes.
Here's the short video of stirring:
http://s242.photobucket.com/albums/f...t=MVI_2105.flv

After that I warmed up the nitration mix with hot water to 30-43°C for around 25 minutes. After warming it up I noticed viscosity of nitration mix drastically dropped. I damped the nitration mixes in around 1.9L of cold distilled water I stirred the whole thing virgously to prevent decomposition of product. I decanted waste water and added fresh one few times then few times with 2% NaHCO3 sollution. And finaly few times with distilled water again.
The picture of percipitate ready to filter:
http://i242.photobucket.com/albums/f...o/IMG_2111.jpg

I filtered the product and let most of the water leak trough filtering paper. I collected the product and dissolved it in 400mL of boiling acetone with teaspoon of NaHCO3 dissolved in it mixture started to foam after it ''foamed out'' it was moved in hot water while stirring to distribute heat. I filtered it and damped the filtrate in NaHCO3 sollution in distilled water while stirring. I decanted most of the liquid, filtered the crystals and dried them for around 2 days on warm pipe 30-40°C.
The product with AA battery for comparison:
http://i242.photobucket.com/albums/f...Picture003.jpg

http://i242.photobucket.com/albums/f...Picture004.jpg
The yield was satisfying IIRC it was around 34g.
Few days later I attempted another synth it was another paralel nitration.
Materials used: 3x (20g of PE, 68mL of 65% HNO3 and 49.5mL of 96% H2SO4)
This time I used three 250mL beakers for stirring I used two thermomethers and glass rod.
I put the beakers in salt/ice bath, mixed the acids at below 5°C. I started adding PE in 1-2g a time while not letting temperature above 20°C. I was stirring constantly (as much as I coud stir 3 beakers). After adding all the PE I left beakers stand for around 10 minutes in room temperature water with occasional stirring.
http://i242.photobucket.com/albums/f...ETN120g004.jpg

I decided to move nitration mix from all three beakers to bigger 2L beaker (It woud be hard to control 3 beakers at once). After most of nitration mix was transferred into the large beaker nitration mix was warmed and kept on 45°C (+-1°C) for 30 minutes.
Nitration mix in 2L beaker at around 45°C
http://i242.photobucket.com/albums/f...ETN120g005.jpg
After heating it was damped in cold distilled water washed couple of times with distilled water and NaHCO3 sollution.
PETN ready to filter: http://i242.photobucket.com/albums/f...o/PETN120g.jpg

I filtered product and dissolved it in 500mL of hot acetone. Recrystalization was done similar way at the synth before acetone sollution was filtered and damped in 5L of distilled water with NaHCO3 dissolved in it. After damping it I decanted liquid and collected crystals on filtering paper.
The product yield was dissapointing around 70g from 60g of PE. I suspect the improper ratio of acids/PE and some of acetone sollution got spilled for souch a bad yield…

This is the product from both above mentioned synths 103.8g of recrystalized PETN:
http://i242.photobucket.com/albums/f...TN1038g004.jpg
PETN flame test: http://rapidshare.de/files/40755178/... test.avi.html

Next thing I tried was a bit bigger nitration. I helped myself PE/acids ratios using some macro. I got those ratios by elevating ammounts of reactants.
That's the screenshot: http://shrani.si/f/19/pV/4FbmS5jp/petn-macro-2.png

Materials used: 100g of PE, 390mL of 65% HNO3, 255mL of 96% H2SO4
First I mixed the acids below 30°C in 2L wide neck beaker which was placed in salt/ice bath. Acid mixing took me couple of hours so as the addition of PE (2-4g a time) while keeping temperature below 10°C. After all PE has been added beaker was left in warm water at 15-20°C for around 50 minutes I occassionally stirred it. Fuming was very intense during the whole procedure and I coudn't stand HNO3 fumes so I used wet cloth as some form of respiratory protection. After 50 minutes beaker was moved in boiling water(water had 80-90°C) I was stirring virgously to prevent localized heating and moving beaker in and out of hot water bath untill I reached temperature of 45°C. I sustained that temperature for around 40 minutes then I damped the whole thing in 5-6L of water and washed it couple more times with distilled water and NaHCO3 sollution. At the end I decanted remaining liquid and filtered the product. After filtering I used some toilet paper to soak even more water from crude PETN.
Then it was dissolved in 1-1.3L of acetone. Acetone sollution was warmed up to it's boiling point and filtered - I managed to filter most of it but then filter paper clogged badly PETN started to recrystalize on it. I became tired of it knowing filtration woud take forever and product loss will be great(it will stay on wrong side of filter paper) so decided damp the rest in distilled water with NaHCO3 dissolved in it. Liquid was once again decanted and crystals

collected on filtering paper.
I got 161.6 g of recrystalized and dry product.
Here are some pictures of synth (nitration and drying):
http://rapidshare.de/files/40755246/...ation.rar.html

And some pictures of dry PETN in 2 piles(265.4g total) smaller pile 103.8g larger pile 161.6g AA batery is placed inbetween as the scale:
http://i242.photobucket.com/albums/f...g?t=1224932251
http://i242.photobucket.com/albums/f...g?t=1224932252

About a month and a half later I detonated 20 grams of PETN/Al
Ratios(by weight of course):
94% PETN (18.8g)
6% Al (1.2g)
Around 0.4grams of HMTD packed in straw was used as a detonator. The charge was packed in plastic cube like box. The charge was put on asphalt.
Video:
http://www.youtube.com/watch?v=EvkAst3zqug
Camera was 15-18m away on some box.

Pictures:
http://i242.photobucket.com/albums/f...g?t=1224933331
http://i242.photobucket.com/albums/f...g?t=1224933334

Plastizicing PETN:

Ratios used:
83.6% PETN (~91,9g)
4.9% Motor oil (~5,4g)
11.5% PIB (~12.7g)
I used 110g of reactants but got only 103g of product (I guess the rest sticked to ''equipment'' I worked with).
Procedure:
PIB (in form of ratglue) was dissolved in around 50-80mL of diethyl ether, motor oil was added to sollution and finaly the PETN after motor oil dissolved. I homogenized the mixture with small wooden stick and I let the solvent evaporate on hot water bath outside.
103g of PETN/PIB/Motor oil
http://i242.photobucket.com/albums/f...g?t=1224935426
Video showing mechanical properties of ''plastique'':
http://rapidshare.de/files/40756625/...N-PIB.avi.html
It was packed in shaped charge a month after manufacture.
Dimensions:
Liner : 50mm spherical shaped glass (lightbulb)
Standoff: 125mm from top of the liner
Some pictures of it:
http://i242.photobucket.com/albums/f...g?t=1224936012
http://i242.photobucket.com/albums/f...g?t=1224936013
http://i242.photobucket.com/albums/f...g?t=1224936014
Unfortunatelly glass broke right before detonation and I detonated it anyway. Here's the detonation video:
http://www.youtube.com/watch?v=TUnMvlJ8plc
And consiquences: Concrete block SC was on got completely shattered other two got moved by the force of blast:
http://i242.photobucket.com/albums/f...g?t=1224937121
http://i242.photobucket.com/albums/f...g?t=1224937122
http://i242.photobucket.com/albums/f...g?t=1224937123

I had another PETN/Al test:
Ratios used:
36.5g of PETN
2.4g of Al powder(german dark)
Charge was placed on carhood.
No video this time(there is one but I don't want to upload it because of some reason)
There's a picture instead:
http://shrani.si/f/3j/Pd/1oHsf7UL/img7728petn.jpg
It was recorded with my friend's camera(open objective with 20s expousure). It's almost better than a video.
Results - pictures:
http://i242.photobucket.com/albums/f...g?t=1224937274
http://i242.photobucket.com/albums/f...g?t=1224937274
http://i242.photobucket.com/albums/f...g?t=1224937275

Approx 3 months later I plasticized some more PETN:
Ratios used:
65g PETN
4.7g PIB
3.1g Motor oil

Procedure (with pictures):
http://i242.photobucket.com/albums/f...g?t=1224937838
First 3.1g of motor oil was added on ceramic plate.
http://i242.photobucket.com/albums/f...g?t=1224937839
4.7g of PIB on paper was put on motor oil.
http://i242.photobucket.com/albums/f...g?t=1224937839
65g of PETN was weighted for purpouse of plasticizing.
http://i242.photobucket.com/albums/f...g?t=1224937841
3.5g of PETN was weighterd for the use in booster.
http://i242.photobucket.com/albums/f...g?t=1224937840

Diethyl ether was added to 4.7g PIB and 3.1g motor oil and stirred with small wooden stick.
http://i242.photobucket.com/albums/f...g?t=1224937848
PETN was added to PIB/Motor oil sollution in diethyl ether.
http://i242.photobucket.com/albums/f...g?t=1224937849
Everything above mentioned was stirred.
http://i242.photobucket.com/albums/f...g?t=1224937869
The dish with materials was put on hot water bath to let solvent (diethyl ether) evaporate.
http://i242.photobucket.com/albums/f...g?t=1224937872
This is the final product…

Around 6 months later…
http://i242.photobucket.com/albums/f...g?t=1224938269
Pressed plastic explosive with hole for booster/detonator in middle, Electric detonator, 3.5g of PETN is on paper, and 7g HMTD in plastic bottle.
The charge was put on few kilograms of bricks and detonated using electrical ignition.

Detonation video:
http://www.youtube.com/watch?v=K93YWRtZRdQ

Consiquences:
http://i242.photobucket.com/albums/f...g?t=1224938279
http://i242.photobucket.com/albums/f...g?t=1224938280
http://i242.photobucket.com/albums/f...g?t=1224938281
This test showed the good shelf life of PETN (it was prepared almost year before blast) and plastic explosive's shelf life(around half a year old). I noticed only some change in mechanical properties of plasticized PETN and smell change(that's one of the reasons it was detonated).

Around a week ago I sucessfully nitrated some more PETN:

Ratios used:
100g PE
390mL 65% HNO_3
255mL 96% H_2SO_4
Procedure:
Both acids were mixed in 1L erlenmeyer flask (may the 2L beaker rest in peace) in salt/ice bath. Temperature when mixing acids was never let above 25°C. Mixing acids took me around 1h 20 min this time I used real gasmask (M40 with NATO filters) not some wet cloth. After mixing acids I let the temperature drop to around -15°C and started adding PE. First I added it in 4-6g ammounts later it was being added in 6-15g ammounts. I never let the temperature rise above 10°C when adding PE(that wasn't that hard because it was cold outside and salt/ice bath was working great). Addition of PE took me around 2 hours. After addition I put the erlenmeyer flask with precursors in warm water temperature of nitration mix has risen to 26°C I left it there for about an hour with occassional stirring. When I moved it out of water temperature dropped to 14°C. I heated liter or two of water to it's boiling point, and immersed erlenmeyer flask with nitration mix into it while constantly stirring to reduce the chance of experiencing runaway nitration.
Here's the picture of mixture being nitrated:
http://i242.photobucket.com/albums/f...g?t=1224940278
Mixture was heated on 45°C(with 2°C deviation). Like always there have been alot of nitric acid fumes. After around 35 minutes the nitration mixture was damped in 6L of normal water, decanted, washed with some more water, decanded again and finaly washed with distilled water. I didn't neutralized it to this point. After it partially dried I sucked as much water as I coud with toilet paper, added some diethyl ether to dry it a bit further. Next day I dissolved it in total 1L of acetone. I split acetone into 2 parts (2x500mL) because I didn't have proper 2L glass vessel.
After putting ~half of PETN in 500mL of acetone inside 1L erlenmeyer flask:
http://i242.photobucket.com/albums/f...g?t=1224941979

Improvised ''filtration device'' (there's around 5L of $NaHCO_3$ sollution in the bucket below):
http://i242.photobucket.com/albums/f...g?t=1224941983
Heating PETN/acetone sollution neutralized with $NaHCO_3$ in erlenmeyer flask in hot water bath(70-80°C):
http://i242.photobucket.com/albums/f...g?t=1224941984
PETN/acetone sollution (with great excess of $NaHCO_3$ on bottom) at it's boiling point(56-58°C):
http://i242.photobucket.com/albums/f...g?t=1224941984
Same purification procedure was done 2 times I used dense cloth as filter because I didn't want to clog filter like the last time… I was constantly stirring water below when filtering PETN sollution.
After all acetone/PETN sollution was filtered:
http://i242.photobucket.com/albums/f...g?t=1224942804
Like most times before I decanted most of liquid PETN percitipated in. <2mm needles formed which broke down into smaller crystals after filtering the product.
Here are pictures of filtered still wet PETN:
http://i242.photobucket.com/albums/f...g?t=1224943571
http://i242.photobucket.com/albums/f...g?t=1224943585
Drying PETN:
Still wet:
http://i242.photobucket.com/albums/f...g?t=1224943912
Partially dried (after 7h on 30-40*C):
http://i242.photobucket.com/albums/f...g?t=1225067678

It took PETN around 27hours to completly dry here are some pictures (AA battery for scale):
http://i242.photobucket.com/albums/f...g?t=1224944046
http://i242.photobucket.com/albums/f...g?t=1224944047
Dry product weight is 159.7g this time.

Notes and comments and improvements suggestions:
At first few synths I was probably being almost too careful(mixing acids at below -10°C etc, adding PE below 0°C etc.). Most recent PETN synthesis I preformed was a bit modified because of lack of resources(money,distilled water,$NaHCO_3$ acetone…). I think important contributing factor to lower yields in synthesis was not dilluting PETN/acetone sollution enough and some of PETN stayed dissolved in the decanted liquid. I will try to improve that by using 10 times as much distilled water as acetone when recrystalizing the product next time. I hope that's the main cause for lower yields than expected anybody else thinks the same?

After six and a half hours of writing this post I hope it will be useful to somebody.

Last edited by NUKE(slo); October 26th, 2008 at 09:35 PM. Reason: I spotted wrong link

QUOTE